THE WETTING OF ALUMINA-SILICA REFRACTORIES BY MOLTEN GLASS AND ITS SIGNIFICANCE

By

JAY EUGENE COMEFORO

B. S., Rutgers University, 1944
M. S., Rutgers University, 1946

AN ABSTRACT OF A THESIS

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN
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ACKNOWLEDGEMENT

The author wishes to express his utmost appreciation for the advice, encouragement, and assistance offered to him by R. K. Hursh, Professor of Ceramic Engineering. Particular thanks are extended to A. I. Andrews, Head of the Department of Ceramic Engineering, for his interest in this work.

The writer is indebted to B. R. Ray and H. S. Gutowsky of the Department of Chemistry and to J. E. Gieseking, Professor of Soil Physics, for their helpful suggestions.

Grateful acknowledgement is also made to G. F. Stockdale of the Department of Ceramic Engineering for his aid in making available temperature measurement and control equipment, and to J. A. Healy for supplying samples of certain refractories.

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INTRODUCTION

The attack of refractories by liquids at high temperatures has been studied extensively. One of the most important factors in glass corrosion, however, has not been generally considered, namely the influence of the wetting tendency of the glass on the refractory.

The object of this investigation was to determine the effect of the wetting tendency, the pore size, and the alumina-silica ratio of the refractory on the extent of the penetration of the glass into the refractory. The term "wetting" of a solid by a liquid has been frequently employed. Wetting is generally defined in terms of the adhesion between the liquid and the solid and the cohesion of the liquid itself. It is usually evaluated by the magnitude of the contact angle formed between the liquid and the solid. In this study the contact angle tended to approach zero as the glass dissolved the refractory. Consequently, instead of determining the equilibrium contact angle, the contact angle existing after various time intervals was noted. This change of the contact angle with time was regarded as significant since it is the controlling factor in the penetration of glass into pores.

EXPERIMENTAL PROCEDURES

The wetting of alumina-silica refractories by a soda-lime-silica glass at 1200° C. was studied by the sessile drop method. The refractory sample, approximately 1 x 1 x ¼ inch, was placed in a horizontal two-inch internal diameter, platinum wound, tube furnace. The furnace and specimen were heated to 1200° C. After holding the refractory at this temperature for a least one hour, a glass drop of 4-5 mm. diameter was placed on the flat 1 x 1 inch face of the sample and photographs of the silhouette formed by the drop and the refractory were taken at time intervals of 1, 2, 5, 10, 20, 30, 60, and 120 minutes. The height and width of the drop were measured from the photographs and the

contact angle was calculated. The contact angle is equal to twice the angle whose tangent is the height divided by half the width of the drop at the refractory surface. The contact angles calculated were found to be in agreement with the results obtained by direct measurement.

The method used for the measurement of the effective pore size of the refractories was a modification of the "pressure displacement method". The refractory samples, approximately 1 x 1 x ¼ inch, was first coated with paraffin wax about all edges. It was then saturated with water according to the method outlined by Washburn and placed in a brass holder which permitted the introduction of a thin layer of water over the test piece. Helium was introduced into the system below the refractory sample and the gas pressure slowly increased until the liquid was displaced from a pore in the specimen. The pressure required to drive out the water was recorded in each case until gas was escaping from approximately ten pores. From these measurements the effective radii of the pores were calculated.

The rate of penetration of molten glass at 1200° C. and of glycerin at room temperature into bodies of 99% alumina of various pore sizes was determined. The rapidity with which molten glass was absorbed by the alumina-silica refractories was investigated by two methods. The agreement between the two was good.

SUMMARY OF RESULTS

This investigation was concerned with a study of the factors controlling the rate of penetration of liquids into porous solids; more specifically, with the influence of the effective pore size of the alumina-silica refractories and of the contact angle formed by a soda-lime-silica glass on the rate of penetration of glass into refractories at 1200° C. A review of the theoretical considerations validating the use of the contact angle as a means of measuring the wetting tendency has been presented. The principles concerning the penetration of liquids and the impor-

tance of the size and shape of the pores were noted. The rate of penetration of the glass into the refractories was evaluated by two methods with good correlation. Further information concerning the penetration of the glass into the refractories was obtained from a study of sections of the glass-refractory interfaces. A break was noticed in the relationship between the rate of penetration and the pore size of 99% alumina bodies with two liquids of markedly different properties. An explanation for this sudden change in the slope of the curve was presented. A satisfactory correlation was made between the contact angle, the pore size, and the rate of penetration of the glass into the refractory.

The following conclusions were drawn from this investigation:

1. Of the possible techniques available for the determination of the contact angle, the sessile drop method was found to be most practical. The results obtained by this method were reproducible to within 3°.

2. With liquids of high viscosity, such as the glass used in this study, the roughness of the refractory surface initially caused the apparent contact angle to be greater than that measured on a smooth surface. With longer time, the cavities in the surface became filled with glass and the apparent contact angle became less than that on a smooth surface.

3. The results of the contact angle study indicated that the order of decreasing wetting tendency of the refractories tested was silica, 99% alumina, dense 99% alumina, dense mullite, super duty, flux block, 50% alumina, and semi silica.

4. Several methods were suggested for the calculation of the effective pore radius. Since the volume of glass entering a refractory was primarily dependent upon the size of the largest pores, it was felt that a knowledge of the size of these pores would be most desirable. For this

reason the "pressure displacement method" was employed. By this means the effective pore radius of the ten largest pores was ascertained.

5. The order of decreasing pore size of the refractories was as follows: 50% alumina, 99% alumina, silica, semi silica, super duty, mullite, flux block, and dense 99% alumina.

6. The petrographic analysis failed to indicate any change in the refractory or the glass at the interface, except for evidences of solution of some refractories by the glass.

7. The resistance of the refractories to the penetration of molten glass was in the order, semi silica, 50% alumina, super duty, flux block, dense mullite, mullite, dense 99% alumina, and lastly silica.

8. A study of the rate of penetration of two different liquids into a series of 99% alumina bodies of different pore sizes showed that a sharp change in the rate of penetration occurred at a pore radius of 10-15 microns. An explanation for this has been presented, based upon the effect of the change in radius on the quantity of liquid which may enter the pores and upon the change in the absorptive capacity of the pores with change in pore radius.

9. A comparison of the relative rates of penetration of glass into a series of refractories may be obtained by multiplying the fourth power of the cosine of the contact angle measured and the cube root of the "average" pore radius as determined by the pressure displacement method. The product thus determined serves to rank the refractories according to their resistance to glass penetration; the lower the result, the more resistant to penetration.

VITA

Jay Eugene Comeforo was born on August 3, 1922, in Staten Island, New York. He received his elementary and high school education in the public school system of that state.

In the fall of 1940 he entered Rutgers University and graduated in January 1944 with the degree of Bachelor of Science in Ceramics. Upon graduation he became a member of the staff as Research Assistant. From January 1945 to June 1946 he was Research Associate and concurrently taught several courses. During this period he became associated with the Bureau of Mineral Research of Rutgers University and was in charge of mineral beneficiation. While on the Ceramic Department staff he matriculated in the Graduate School, and in February 1946 he completed the requirements for the degree of Master of Science in Ceramics.

He accepted the position of Special Research Assistant in Ceramic Engineering at the University of Illinois in July 1946 and held this position until June 1947 when he was made Special Research Associate. In February 1948, in order to hasten progress toward the doctorate, he accepted the Illinois Clay Products Company Fellowship.

He is a member of the American Ceramic Society, the Mineralogical Society of America, Sigma Xi, and Keramos.

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